# Control Stability of a Heat-Flow-Meter Apparatus

## ROBERT R. ZARR

Building and Fire Research Laboratory National Institute of Standards and Technology Gaithersburg, MD 20899

ABSTRACT: Calibration measurements of a commercial heat-flow-meter apparatus are presented. The apparatus has been calibrated using the same specimen of high-density fibrous-glass board over a period of four years, from 1989 to 1993. Seventy-three tests have been conducted, generally at ambient conditions of 24°C with a moderate temperature difference of either 15, 22 or 27°C across the specimen. Variations within a set of data for each test have been examined to verify underlying assumptions of randomness, normal frequency distribution of errors, repeatability, and stability of the data. Variations between test data indicate a small drift, on the order of one percent over four years, in the calibration factor of the apparatus. A model has been developed to describe the small drift with time. The analysis of variations between test data has also identified intermittent shifts in the precision of the calibration factor of the apparatus.

## 1. INTRODUCTION

THE STABILITY PROBLEM can be re-stated as follows: is the heat-flow-meter apparatus "in statistical control"? That is, are the calibration factors (S) "in statistical control"? To ascertain the answer, a series of calibration tests are conducted and the resulting data analyzed for stability and consistency. If the answer is yes, the process is predicted by the equation: S = C + e, where C is a constant and e is random error. This equation assumes that the process is unbiased; in other words, systematic error is not present. If the

Reprinted from J. THERMAL INSUL. AND BLDG. ENVS. Volume 18 - October 1994

This paper was originally presented at the American Society for Testing and Materials C-16 Workshop on Measurement Errors and Methods of Calibration of Heat-Flow-Meter Apparatus at Williamsburg, VA on April 9, 1994.

answer is no, systematic error is present. Under certain circumstances, it is possible to identify the anomaly(ies) perturbing the process. In this case, the process is predicted by S = f(t) + e, where f(t) is some time-dependent function. In the worst case, the process is unpredictable and the process, including the apparatus, may have to be re-evaluated and possibly revised.

The issue of control stability is essential when calibrating a heat-flow-meter apparatus. In our case, the assessment of control for this apparatus includes two components—local variations (within-test stability for each of the 73 tests) and global consistency (between-test stability across the 73 tests). This paper presents a description and analysis of the experimental data. The analysis includes a brief background on the stability criteria for an ideal process and examines the within and between variations of the test data. A description of the heat-flow-meter apparatus has been published previously [1]. Descriptions of similar apparatus are presented elsewhere [2].

# 2. EXPERIMENTAL

From 1989 to 1993, 73 calibration tests were conducted with the heat-flow-meter apparatus using the same specimen of high-density fibrous-glass board. The calibration factor, S, (W/m²·mV) for the steady-state portion of each test was determined by:

$$S = \frac{\sum_{i=1}^{n} \Delta T_{i}}{\sum_{i=1}^{n} E_{i} \left(\frac{L}{\lambda}\right)}$$
 (1)

Here,  $\Delta T_i$ , the temperature difference between the hot and cold surfaces, and  $E_i$ , the output of the heat-flux transducer, were determined at each scan interval, *i*. The thickness, L, of the specimen was measured independently with vernier calipers. The thermal conductivity,  $\lambda$ , was predicted by an equation developed by Siu [3]:

$$\lambda = 0.02052 + 1.303 \times 10^{-5} \varrho + 4.015 \times 10^{-10} T_m^3 \tag{2}$$

where  $\varrho$  and  $T_m$  were the specimen bulk density (kg/m³) and mean temperature (K), respectively. For this analysis, individual observations,  $S_i$ , of the calibration factor were re-computed for each test to assess local variations.

Table 1. Breakdown of data.

|                  | Number of Tests |      |      |      |      |                |      |      |      |
|------------------|-----------------|------|------|------|------|----------------|------|------|------|
|                  | T <sub>m</sub>  |      | ΔΤ   |      |      | T <sub>e</sub> |      |      |      |
| Test<br>Operator | 13°C            | 24°C | 15°C | 22°C | 27°C | 22°C           | 60°C | 80°C | 90°C |
| 1                | 0               | 52   | 0    | 41   | 11   | 3              | 1    | 3    | 45   |
| 2                | 0               | 7    | 0    | 7    | 0    | 0              | 0    | 1    | 6    |
| 3                | 0               | 7    | 0    | 7    | 0    | 0              | 0    | 0    | 7    |
| 4                | 0               | 3    | 0    | 3    | 0    | 0              | 2    | 0    | 1    |
| 5                | 0               | 2    | 0    | 2    | 0    | 0              | 2    | 0    | 0    |
| 6                | 0               | 1    | 0    | 1    | 0    | 0              | 1    | 0    | 0    |
| 7                | 1               | 0    | 1    | 0    | 0    | 0              | 0    | 0    | 1    |
| Totals           | 1               | 72   | 1    | 61   | 11   | 3              | 6    | 4    | 60   |

The observations were then averaged to obtain S, or:

$$S_{i} = \frac{\Delta T_{i}}{E_{i} \left(\frac{L}{l}\right)} \to S = \sum_{i=1}^{n} \frac{S_{i}}{n}$$
(3.4)

The differences in values of S computed from Equations (1) and (4) were less than 0.01%.

The principal parameter of interest for the stability analysis was time, t. However, over the course of four years, other parameters were (inadvertently) introduced that affected the calibration of the apparatus. These parameters included, among others, mean temperature  $(T_m)$ , temperature difference  $(\Delta T)$ , and conditioning temperature  $(T_{\bullet})$  of the specimen prior to the test. The breakdown of the data (i.e., frequency of occurrence) is summarized in Table 1. As noted in Table 1, the experimental design was not balanced with respect to  $T_m$ ,  $\Delta T$ , or  $T_{\bullet}$ . A balanced design would require an equal number of runs for each parameter by each operator.

## 3. ANALYSIS OF DATA

# 3.1 Background

An ideal process in a state of statistical control exhibits four characteristics:

1) the location of the data is fixed (i.e., the mean does not change with time);

2) the variation of the data is fixed and is preferably low (i.e., the standard deviation does not change with time);

3) the data are random, that is, adja-

cent data "behave" independently of one another; and 4) the frequency distribution of the data is fixed (not changing with time) and, preferably, normally distributed. The benefit of a process that is in a state of statistical control is predictability—the ability to predict where the process will be in the future. As mentioned, the model that describes such an ideal process is S = C + e.

## 3.2 Within-Set (Local) Variations

The variations of observations (that is, calibration factor,  $S_i$ ) within each set of test data were analyzed graphically to verify the underlying assumptions of stability (fixed location and variation), randomness, and normality. A four-step method was applied:

- 1. a run-sequence plot  $(S_i \text{ versus } i)$  checks for systematic and random changes
- 2. a lag plot  $(S_i \text{ versus } S_{i-1})$  checks for randomness
- 3. a histogram (of  $S_i$ )—checks the frequency distribution
- 4. a normal probability plot (of  $S_i$ ) checks the normality assumption

This four-step analysis was executed for each of the 73 calibration tests. The analyses for two calibration tests, 29 and 8, are illustrated in Figures 1 and 2 respectively, and summarized in Table 2. For most of the 73 data sets, the underlying assumptions were satisfied and thus the set of data deemed "in control." Figure 1 shows the analysis for test 29 and illustrates an ideal "in control" process. Here, the location and variation [Figure 1(a)] are "fixed" (not changing with time). The lag plot [Figure 1(b)] has no discernable structure and appears as a "bull's-eye." The histogram [Figure 1(c)] is bell-shaped; and the (normal) probability plot [Figure 1(d)] is linear. The test statistic known as the normal probability correlation coefficient (NPCC) is discussed elsewhere [4], but its graph is easy to interpret; if linear, then normal.

The data from a few other tests were not normally distributed and, in fact, suggested an underlying bimodal mechanism. This mechanism is illustrated in the data of test 8 (Figure 2). Test 8 was the only test that failed to satisfy

Table 2. Summary of within-set assumption for tests 29 and 8.

| Criteria                  | 29  | 8   |  |
|---------------------------|-----|-----|--|
| 1. Fixed location?        | Yes | Yes |  |
| 2. Fixed variation?       | Yes | No  |  |
| 3. Randomness assumption? | Yes | No  |  |
| 4. Normality assumption?  | Yes | No  |  |

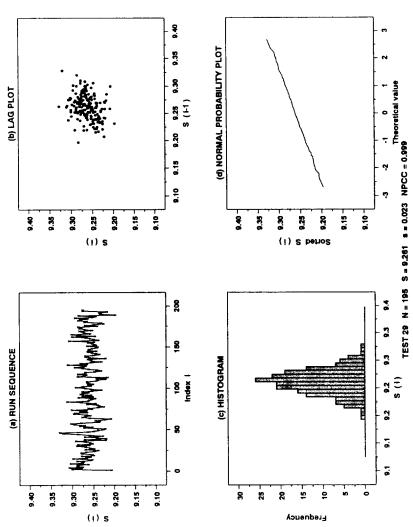


FIGURE 1. Local (within-set) variations of test 29. (a) run-sequence plot, (b) lag plot, (c) histogram, (d) normal probability plot (index of normality).

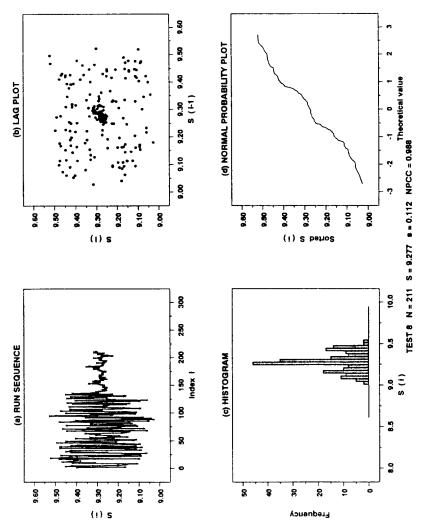


FIGURE 2. Local (within-set) variations of test 8. (a) run-sequence plot, (b) lag plot, (c) histogram, (d) normal probability plot (index of normality).

the assumption of fixed variation [Figure 2(a)]. After further investigation, the source of variation was traced to the output of the heat-flux transducer. As noted in Figure 2a, the data falls into two groups. Although the means for both groups of observations were essentially the same, the standard deviations were different by a factor of about 8. This behavior was not symptomatic of the transducer itself, but instead, of some external electrical contamination. At present, the external contamination has not been identified.

# 3.3 Between-Set (Global) Consistency

The same questions concerning changes in variation, location, as well as normality were examined globally by checking consistency between the 73 sets of test data. A graphical compilation of the 73 tests is illustrated in Figure 3. Figure 3(a) shows a run sequence of means where S is plotted against test number (1 to 73, inclusive). Included with each point are error bars of twice the standard deviation of each mean. Three characteristics in the data were observed: 1) a downward drift of about one percent in the means; 2) a large variability for test 8 (discussed above); and, 3) small (neglecting test 8) changes in the standard deviations. As noted in the runsequence plot of standard deviations [Figure 3(b)], the changes were actually small intermittent shifts. The source of these shifts was traced to changes in the output of the heat-flux transducer as discussed above. The index of normality [Figure 3(c)] indicated that about two-thirds of the test data were normally distributed (coefficients ≈ 1). Further analysis is required to explain non-normal data sets. The number of observations per test is shown in Figure 3(d).

## 4. MODEL FOR GLOBAL DATA

A time-sequence plot of the means (S versus t) is shown in Figure 4. The initial time, t=0, denotes the apparatus's return from the manufacturer after renovation, which included installation of: 1) a new heat-flux transducer; 2) new digital temperature controllers; and 3) new type-K\* thermocouples [1]. Since the apparatus was essentially new, most of the data was collected within the first half-year to verify the operation of the apparatus. As time progressed, the calibration tests were conducted at greater time intervals.

If the apparatus was "in control" for all 73 tests, then the appropriate model would be: S = C + e. From Figures 3(a) and 4, it is clear this was not the case. Instead, there was a downward drift. To account for the down-

<sup>\*</sup>Nickel and 10 percent chromium versus nickel and 5 percent aluminum and silicon.

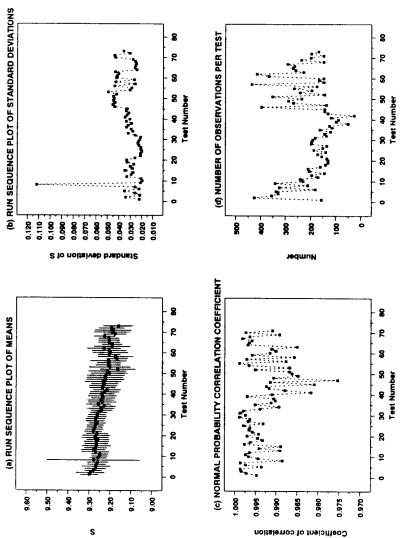


FIGURE 3. Global compilation of test data. (a) run-sequence plot of means, (b) run-sequence plot of standard deviations, (c) run-sequence plot of "index of normality," (d) number of observations per test.

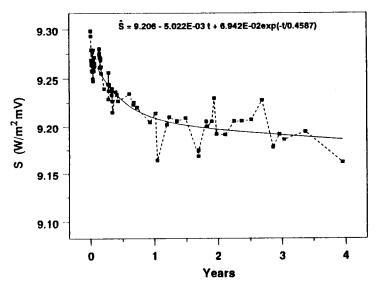


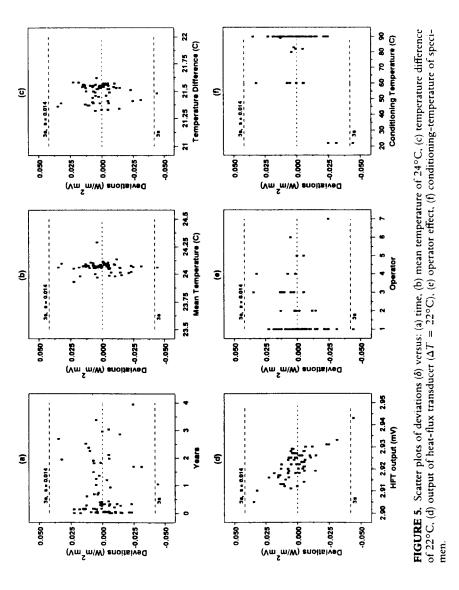
FIGURE 4. Time-sequence plot of means and corresponding ("drift") model.

ward drift in the means with time (t, in years), the following model (constant + linear + exponential) was developed:

$$\hat{S} = 9.206 - 5.022 \times 10^{-3} t + 6.942 \times 10^{-2} \exp\left(\frac{-t}{0.4587}\right)$$
 (5)

This fitted model is shown in Figure 4 as a solid line. The model assumes that errors were random. In practice, however, errors may be from non-random assignable causes. Identifying the causes provides opportunity for refining and improving the model as discussed below.

The adequacy of the model was examined by plotting the deviations  $(\delta = S - \hat{S}, \text{ in } W/m^2 \cdot \text{mV})$  from the model versus 1) time; 2) mean temperature of 24°C; 3) temperature difference of 22°C; 4) output of the heat-flux transducer; 5) operator; and 6) conditioning temperature. These plots are shown in Figures 5(a), (b), (c), (d), (e) and (f) respectively. Control limits of three standard deviations ( $s = 0.014 \ W/m^2 \cdot \text{mV}$ ) of the deviations ( $\delta$ ) are shown as dashed lines. Note that a few test points are close to the limits and one is outside. Figures 5(a), (b) and (c) do not indicate any trends in the deviations, signifying a satisfactory fit. A systematic trend is noted, however, in the deviations versus the output of the heat-flux transducer for  $\Delta T = 22^{\circ}$ C [Figure 5(d)]. This effect has been studied recently by Lackey et al. [5] for a similar heat-flow-meter apparatus and the reader is referred to



that paper for further details. Other systematic trends are noted in Figures 5(e) and (f), particularly at a conditioning temperature of 22°C. In order to conclude which of these parameters affected the calibration, confounding effects must be eliminated by conducting a balanced-design experiment.

#### 5. SUMMARY

Over a period of four years, 73 calibration tests of a heat-flow-meter apparatus were conducted with a single specimen of fibrous-glass board. Most tests were conducted at 24°C with either a temperature difference of 15, 22 or 27°C. An analysis of local variations of the test observations indicated that most of the sets of data were in a state of statistical control. There were, however, small intermittent shifts in the repeatability (within-laboratory precision) of the calibration factor due to changes in the output of the apparatus's heat-flux transducer. A global check of the test data revealed a small downward drift, on the order of one percent, in the calibration factor over the four-year period. A model of the form:  $S = a_0 + a_1t + a_2 \exp(t/a_3)$  was fit to the data to account for the drift with time (t). An analysis of the deviations from the model indicated further improvement in the model could be obtained by considering other factors, such as the output of the heat-flux transducer, operator, and pre-conditioning temperature of the specimen.

## 6. ACKNOWLEDGEMENTS

The author appreciates discussions with Dr. Jim Filliben of the Statistical Engineering Division of NIST. The author also acknowledges the following students who assisted with the data collection and preparation of the manuscript: John Judge, Kevin Simmons, Mike Price, Dan Shapiro, Chris Saunders, Ken Kramp, and Holly Chandler.

## REFERENCES

- 1. Zarr, R. R. and B. A. Licitra. 1991. "Calibration at 24°C of a Heat-Flow-Meter Apparatus Having 610 mm Square Plates," NISTIR 4539 (May).
- 2. ASTM. 1991. "Standard Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus," ASTM C518-91, Annual Book of ASTM Standards, American Society for Testing and Materials, Philadelphia.
- 3. Siu, M. C. I. 1980. "Fibrous Glass Board as a Standard Reference Material for Thermal Resistance Measurement Systems," ASTM STP 718, Thermal Insulation

- Performance, D. L. McElroy and R. P. Tye, eds., Philadelphia, American Society for Testing and Materials, pp. 343-360.
- 4. Filliben, J. J. 1975. "The Probability Plot Correlation Coefficient Test for Normality," *Technometrics*, 17:111-117, (February).
- 5. Lackey, et al. 1994. "Calibration of the Heat Flow Meter Apparatuses at the NRCC," *Journal of Thermal Insulation* (this publication).